

# REPORT DOCUMENTATION PAGE

AFRL-SR-AR-TR-04-

Public reporting burden for this collection of information is estimated to average 1 hour per response, including the time for reviewing instructions, gathering existing data needed, and completing and reviewing this collection of information. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing this burden, to Washington Headquarters Services, Directorate for Information Operations and Reports (0704-0188), 4302. Respondents should be aware that notwithstanding any other provision of law, no person shall be subject to any penalty for failing to comply with a collection of information if it does not have a valid OMB control number. PLEASE DO NOT RETURN YOUR FORM TO THE ABOVE ADDRESS.

1. REPORT DATE (DD-MM-YYYY)

Dec 10<sup>th</sup> 2003

2. REPORT TYPE

Final

3. DATES COVERED (From - To)

Apr 01 2002 to Apr 01 2003

4. TITLE AND SUBTITLE

Synthesis of Oxide Fibers Using a Fiber Drawing Machine

5a. CONTRACT NUMBER

5b. GRANT NUMBER

F49620-02-1-0285

5c. PROGRAM ELEMENT NUMBER

5d. PROJECT NUMBER

5e. TASK NUMBER

5f. WORK UNIT NUMBER

6. AUTHOR(S)

Professor Waltraud M. Kriven

20040130 081

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)

Materials Science and  
Engineering Dept., University  
of Illinois at Urbana-  
Champaign,

1304 W. Green St.,  
Urbana IL 61801  
Tel: (217) 333 5258  
Fax: (217) 333 2736  
Email: kriven@uiuc.edu

8. PERFORMING ORGANIZATION REPORT  
NUMBER

9. SPONSORING / MONITORING AGENCY NAME(S) AND ADDRESS(ES)

Dr. Joan Fuller  
Ceramic and Non-Metallic  
Materials Program Manager  
Directorate of Aerospace and  
Materials Engineering

Air Force Office of Scientific  
Research  
4015 Wilson Blvd, room 713  
Arlington, VA 22203-1977  
Ph. (703) 696 7236

10. SPONSOR/MONITOR'S ACRONYM(S)

11. SPONSOR/MONITOR'S REPORT  
NUMBER(S)

12. DISTRIBUTION / AVAILABILITY STATEMENT

Unlimited

13. SUPPLEMENTARY NOTES

14. ABSTRACT

The Marksman Fiber Drawing Machine, (Chemat Technology Inc., Northridge, CA) is a research scale, positive displacement, piston-type, fiber extruder. The machine was custom made to extrude various compositions of fibers from polymeric precursors specifically designed with sufficient flexibility to cover anticipated research requirements. It has a capacity of 10-150 grams, offers operational temperatures up to 500°C, a 10 – 10,000 m/min take up capacity, and 2400 Watts heater capacity. It allows the continuous drawing of fiber while the diameter of the spinnerette hole can be designed to fit application requirements. Standard spinnerette orifice diameters of 200 nm, 150 nm and 100 nm were supplied. In addition, an orifice diameter of 30 nm was custom made for research use. The machine for UIUC was custom made with an integrated high temperature furnace to allow the fiber to be dried and sintered at elevated temperatures, in three zones, up to 1,200°C with programmable controls.

15. SUBJECT TERMS

Fiber extruder, polycrystalline powders, 200nm to 30 nm orifice

16. SECURITY CLASSIFICATION OF:

none

a. REPORT

b. ABSTRACT

c. THIS PAGE

17. LIMITATION  
OF ABSTRACT

18. NUMBER  
OF PAGES

4

19a. NAME OF RESPONSIBLE PERSON  
Prof. Waltraud Kriven

19b. TELEPHONE NUMBER (include area  
code)  
(217) 333 5258

### Description of Instrument

The Marksman Fiber Drawing Machine, (Chemat Technology Inc., Northridge, CA) is a research scale, positive displacement, piston-type, fiber extruder. The machine was custom made to extrude various compositions of fibers from polymeric precursors specifically designed with sufficient flexibility to cover anticipated research requirements. It has a capacity of 10-150 grams, offers operational temperatures up to 500°C, a 10 – 10,000 m/min take up capacity, and 2400 Watts heater capacity. It allows the continuous drawing of fiber while the diameter of the spinnerette hole can be designed to fit application requirements. Standard spinnerette orifice diameters of 200 nm, 150 nm and 100 nm were supplied. In addition, an orifice diameter of 30 nm was custom made for our research use. The machine for UIUC was custom made with an integrated high temperature furnace to allow the fiber to be dried and sintered at elevated temperatures, in three zones, up to 1,200°C with programmable controls. Fig. 1 shows the complete fiber extruder assembled in our laboratory. Fig. 2 focuses on the take up spool at the foot of the stand and beneath the heating furnace.

### Status of Extruded Fibers

Polycrystalline mullite monofilaments were prepared by the extrusion method. Extrusion was carried out in our new fiber extruder. The orifice for extrusion was varied from 30  $\mu\text{m}$  to 250  $\mu\text{m}$ . Hydrothermally grown KM mullite powder (KM Mullite 101, Kyoritsu Ceramic Materials Co. LTD., Nagoya, Japan) was used as starting powder. The composition of the powder consisted of 60 mol%  $\text{Al}_2\text{O}_3$  and 40 mol%  $\text{SiO}_2$  and the average particle size was 0.77  $\mu\text{m}$ .

PVA and methyl cellulose were used as a binder for extrusion. 15 wt% of PVA (Polyvinyl Alcohol, Airvol 540S, Celanese Chemicals, USA) solution in DI water was prepared as PVA binder. Also 3 wt% of cellulose (Hydroxypropyl Methyl Cellulose, Sigma-Aldrich, Milwaukee, WI) binder was added. The prepared mullite powder was mixed with the organic binder to create a paste for extrusion. The mixed paste was loaded in the fiber extruder and mullite fiber was extruded. The speed of the piston during extrusion was controlled in order to form a uniform diameter of fiber (Fig. 3). The extruded fibers were dried in air and sintered at various temperatures up to 1600 °C. The microstructure of sintered fiber was characterized by scanning electron microscopy (Zeiss DSM960 SEM).

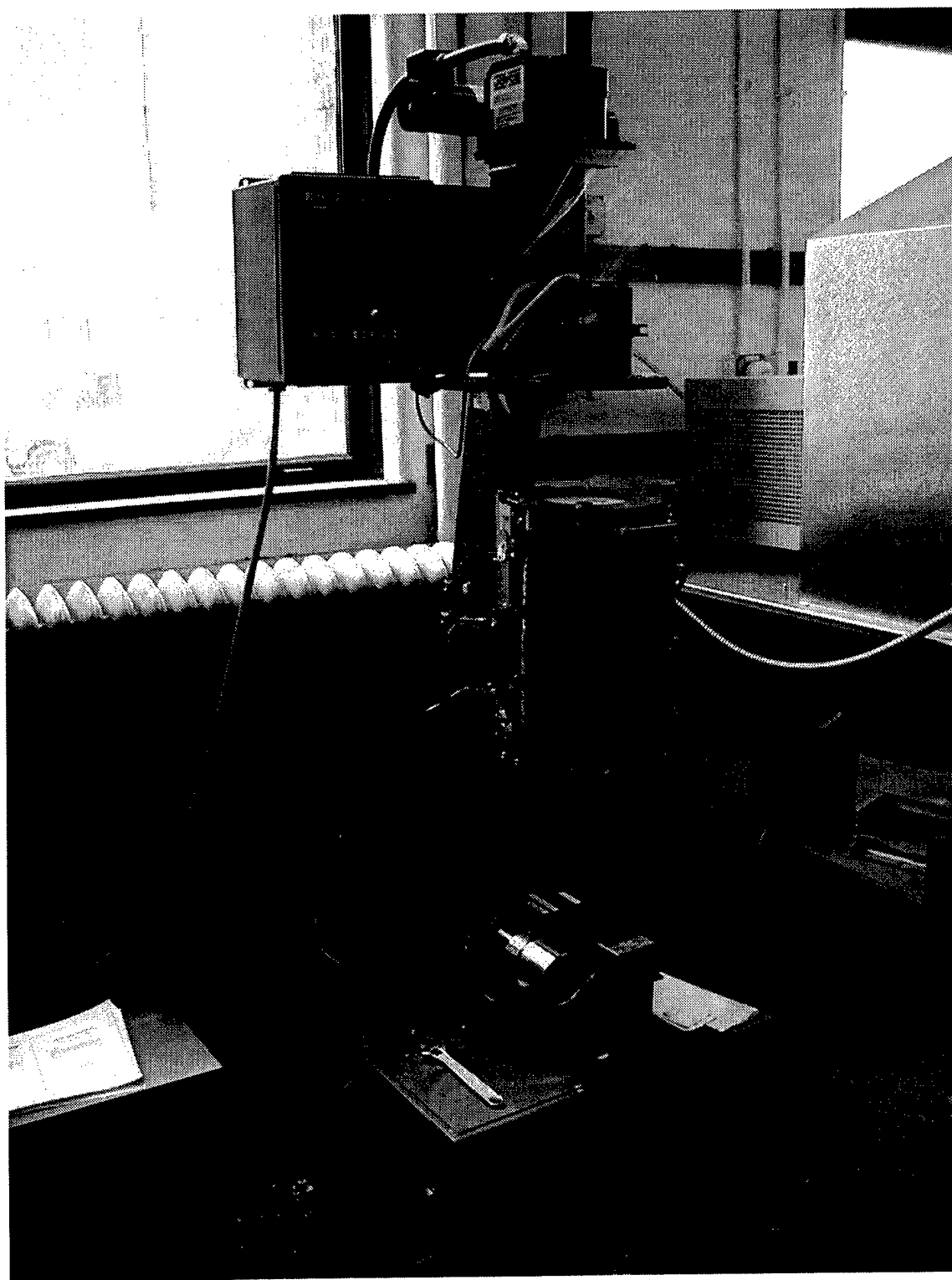


Fig. 1 Photograph of complete Marksman fiber extruder set up at UIUC

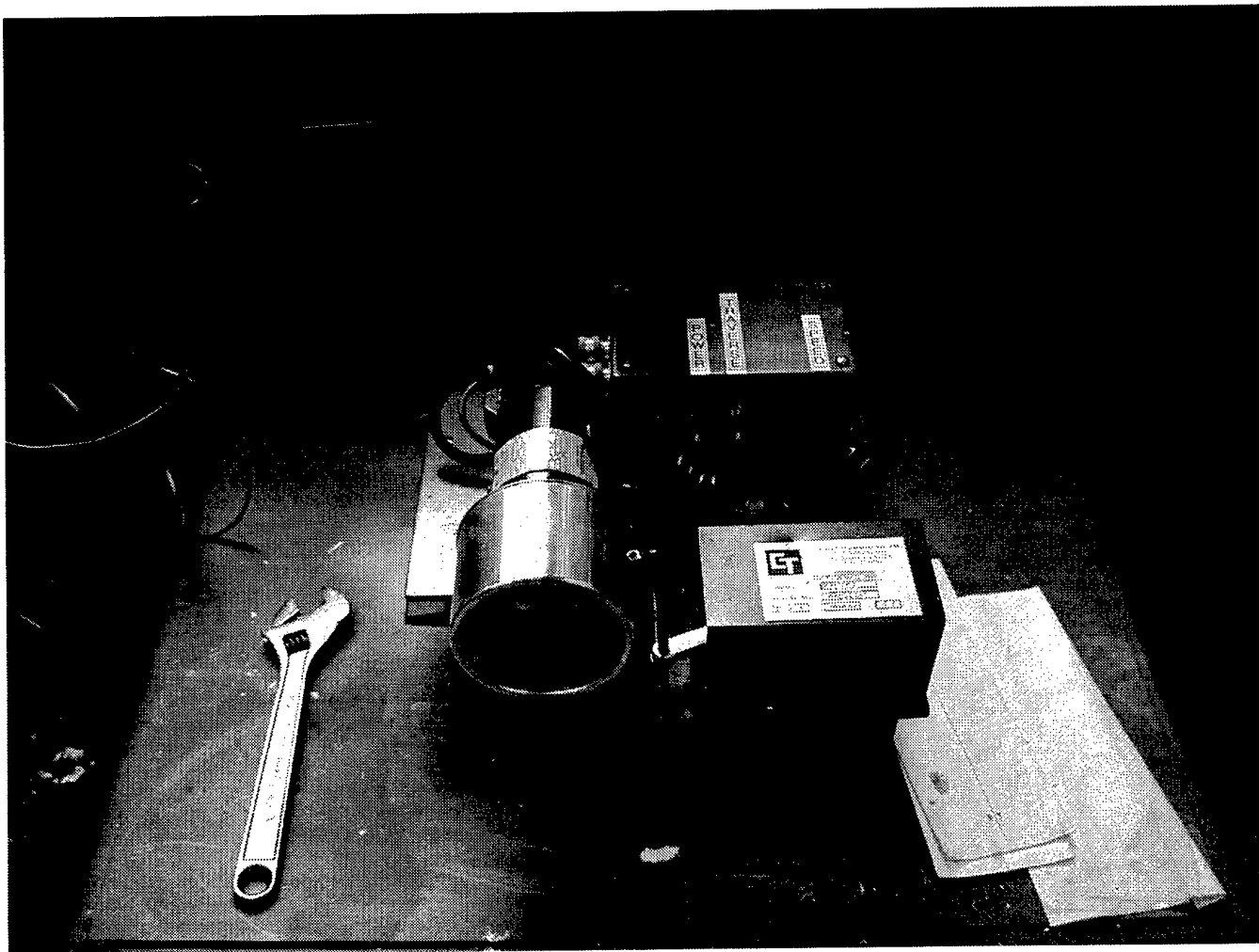


Fig. 2. The take up spool of the Marksman fiber extruder

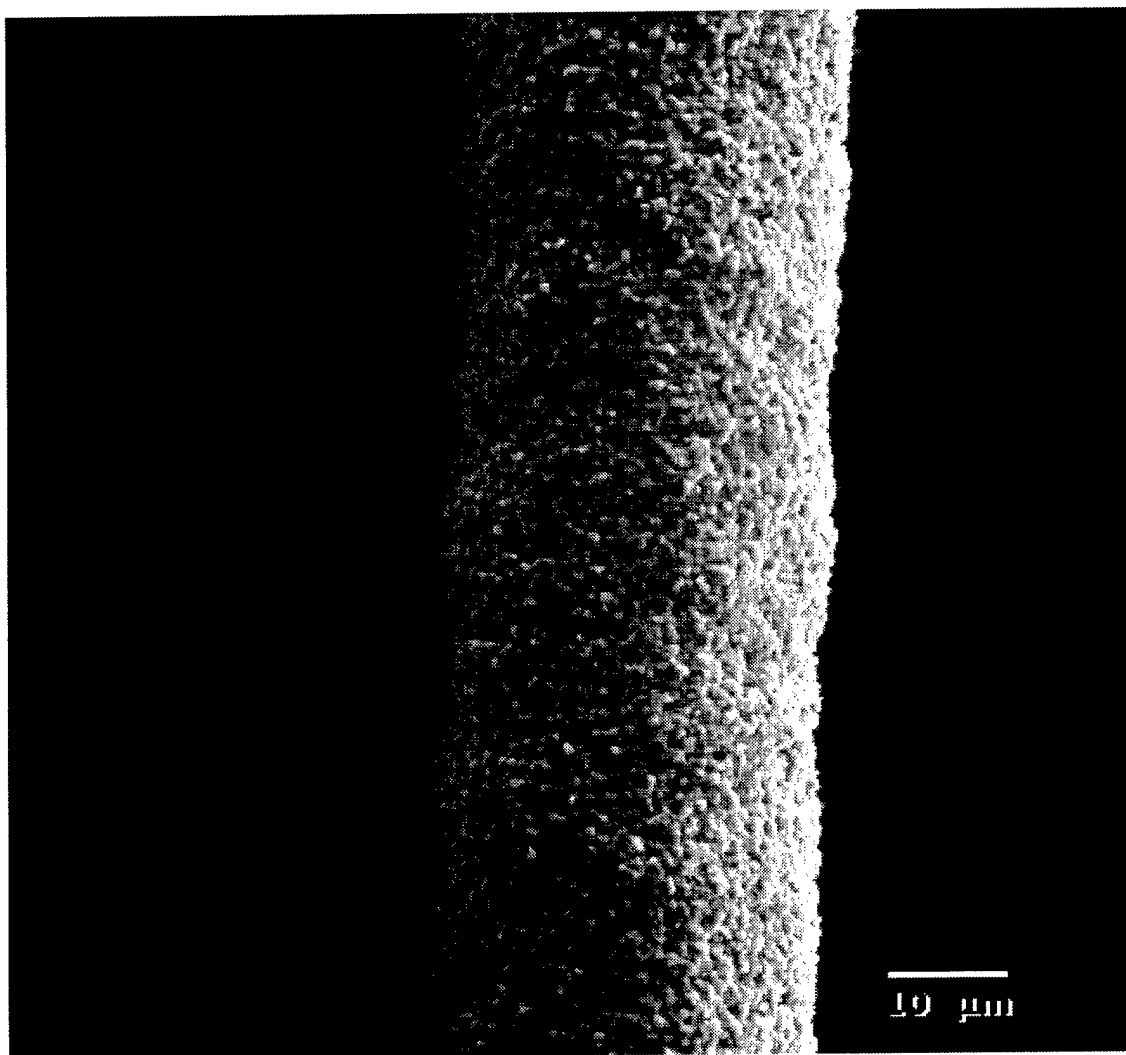


Figure 3 Mullite fiber drawn with the Marksman extruder and sintered at (1600 °C/1hr). This fiber will now be passed through the quadrupole furnace at an optimized temperature and traverse rate.